To: Liaisons

From: Nabil Yacoub
Department Lead Liaison

Date: August 7, 1996

Subject: **Common Laboratory Contaminants**

ISSUE: Reviewers of data packages have, on various occasions, encountered different levels of volatile and semivolatile compounds reported by some RP labs as laboratory contaminants. In some instances, the compounds were not detected in the blanks. The issue is the identity of the compounds, volatile and semivolatile, that ECL would consider as common lab contaminants, and whether field sampling equipment are contributing to the problem.

ECL RESPONSE: (based on discussions in the ECL's SOP Committee)

The USEPA-CLP [1] has identified the following compounds as common lab contaminants detected in the analysis for volatile and semivolatile organics:

CLP - Volatiles	CLP - Semivolatiles
Methylene Chloride	Common Phthalate Contaminants
Acetone	
2-Butanone	

ECL concurs, and further identifies the following Phthalates as possible lab contaminants:

bis-2 Ethylhexyl Phthalate	n-Butyl Phthalate
Diethyl Phthalate	n-Octyl Phthalate
Benzyl Phthalate	

ECL and other laboratories have also identified Chloroform as a contaminant when analyzing for volatile organic compounds.

When analyzing samples, such as drinking water, for low levels of metals, Si and Al are often

detected as common contaminants absorbed from the air.

To determine the existence and magnitude of contamination resulting from laboratory (or field) activities, the CLP requires laboratories to analyze a number of lab and field **BLANKS.** CLP then sets criteria for the evaluation of data, and specifies actions to be taken when certain levels of contaminants are detected. The Venn diagram (Figure 1. Below) [2] show some blanks which may be used in the sampling/analytical process. An equipment blank, for example, which is intended to measure the cleanliness of the sampling equipment, could potentially be contaminated in the field, during transportation to the lab, or in the laboratory itself. A method blank, on the other hand, could only be contaminated during sample preparation and analysis in the lab.

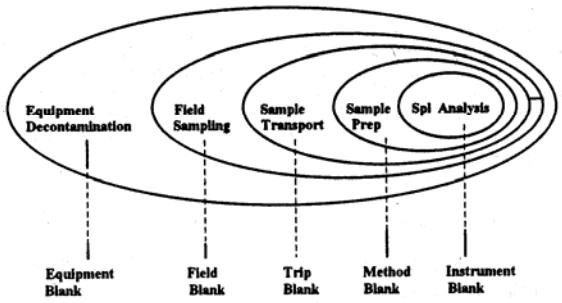

Figure 1 - Blank Samples and Artificially Introduced Contaminants

CLP requires laboratories to apply a set of specific criteria when contaminants are detected in the blanks. Consultants should consider these criteria before releasing a data package. Reviewers of data packages should also be aware of the criteria when reviewing the packages. Information included in the attached excerpts from the CLP National Functional Guidelines for Organic Data Review [1] detail the objectives, criteria, evaluation, and action to be taken in the analysis of volatile and semivolatile organics by CLP laboratories (Attachments).

In general, action regarding unsuitable blank results depends on the circumstances and origin of the blank. Positive volatile and semivolatile sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10 x) the amount in any blank for the common laboratory contaminants, or 5 times (5x) the amount for other target compounds. In instances where more than one blank is associated with a given sample, qualification should be based on a comparison with the associated blank having the highest concentration of a contaminant. The results must NOT be corrected by subtracting any blank value. However, all sample data should be qualified when possible sample contamination might have occurred.

In the inorganic analysis for metals, the 5x criteria apply. If sample results are greater than the instrument Detection Limit (IDL) but less than 5 times the amount found in any blank, results should be qualified as (U). The value is either the sample quantitation limit or the sample detection limit [3].

Sources of contamination may vary laboratory solvents and water, and powdered gloves to lab and field equipment. Analysis of lab and field blanks may help identify these sources of contamination.

Professional judgment is essential particularly when reviewing problematic data packages. Blank and sample data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

If you encounter a problematic data package, you may solicit the expert help of ECL staff to provide the needed evaluation. This service has been frequently used by project managers from various activities especially from the Office of Military Facilities (OF). P.A. and Site codes, and a completed Work Form would be required.

Contact Myrto Petreas at (510) 540-3624 or mpetreas@dtsc.ca.gov.

References:

(1) USEPA-CLP Program, National Functional Guidelines for Organic Data Review, February 1994.

(2) USEPA-Region IX, RCRA Corrective Action, Data Review Guidance Manual, July, 1995.

(3) USEPA-CLP Program, National Functional Guidelines for Inorganic Data Review, February 1994.

Attachments

cc: Bart Simmons, Ph.D.
Bob Stephens, Ph.D.

V. Blanks

A. Review Items: Form I VOA (Form I LCV), Form IV VOA (Form IV LCV), chromatograms, and quantitation reports.

B. Objective:

The purpose of laboratory (or field) blank analysis is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, trip blanks, and equipment blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the area, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blanks.

2. A method blank analysis must be performed after the calibration standards and once for every 12-hour time period beginning with the injection of BFB.

3. The method blank must be analyzed on each GC/MS system used to analyze sample for each type of analysis, i.e., unheated purge (water and medium level soil) and heated purge (low level soil).

4. A storage blank must be prepared upon receipt of the first samples from an SDG, and stored with samples until analysis. The storage blank must be analyzed once per SDG.

5. An instrument blank must be analyzed after any sample that has saturated ions from a given compound to check that blank is free of interference and the system is not contaminated.

D. Evaluation:

1. Review the results of all associated blanks on the forms and raw data (chromatograms and quantitation reports) to evaluate the presence of target and non-target compounds in the blanks.

2. Verify that a method analysis has been reported per matrix, per concentration level, for each 12-hour time period on each GC/MS system used to analyze volatile samples. The reviewer can use the Method Blank Summary (Form IV VOA/form IV LCV) to identify the samples associated with each method blank.

3. Verify that a storage blank has been analyzed and included with each SDG and that the storage blanks are free of contamination.

4. Verify that the instrument blank analysis has been performed following any sample analysis where a target analyte(s) is/are reported at high concentration(s).

Action:

If the appropriate blanks were not analyzed with the frequency described in Criteria 2,3, and 4, and 5 then the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be noted for TPO action.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the common volatile laboratory contaminants (methylene chloride, acetone, and 2-butanone), or 5 times (5x) the amount for other volatile target compounds. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must <u>not</u> be corrected by subtracting any blank value.

Specific actions are as follows:

1. If a volatile compound is found in a blank but not found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action.

2. Any volatile compound detected in the sample (other than the common volatile laboratory contaminants), that was also detected in any associated blank, is qualified if the sample concentration is less than five times (5x) the blank concentration. The quantitation limit may also be elevated. Typically, the sample CRQL is elevated to the concentration found in the sample. The reviewer should use professional judgment to determine if further elevation of the CRQL is required. For the common volatile laboratory contaminant the results are qualified by elevating the quantitation limit to the concentration found in the sample when the sample concentration is less than 10 times (10x) the blank concentration.

   The reviewer should note that blanks may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" and "10x" criteria, such that a comparison of the total amount of contamination is actually made.

   Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, he/she should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. In this case, the "5x" or "10x" rules may not apply;
   The target compound should be reported as not detected, and an explanation of the data qualification should be provided in the data review narrative.

3. If gross contamination exists (i.e., saturated by GC/MS), all affected compounds in the associated samples should be qualified as unusable(R) due to interference. This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

4. If inordinate number of other target compounds are found at low levels in the blank(s), it may be indicative of a problem and should be noted for TPO action.

5. The same consideration given to the target compounds should also be given to Tentatively Identified Compounds (TICs), which are found in both the sample and associated blank(s). (See VOA Section XII for TIC guidance.)

6. If contaminants are found in the storage blanks, the following action is recommended.

   a. The associated method blank data should be reviewed to determine if the contaminant(s) was also present in the method blank. If the analyte was present at a comparable level in the method blank, then the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.

      If the analyte was not present in the method blank, then the source of contamination may be in the storage and all associated samples should be considered for possible cross-contamination.

   b. If the storage blank contains a volatile TCL compound(s) at a concentration greater than the CRQL, then all positive results for that compound(s) should be qualified with “J”. If the concentration level in the blank is significantly high, then positive sample results may require rejection and be qualified with “R”. Non-detected volatile target compounds should not require qualification unless the contamination is so high that it interferes with the analysis of the non-detect compounds.

7. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument cross-contamination has affected any positive compound identification(s). If instrument cross-contamination is suspected of having an effect on the sample results.

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Example 1: Sample result is greater than the Contract Required Quantization Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	7	7
CRQL	5	5
Sample Result	60	30

Final Sample Result	60U	30U

In the example for the "10x" rule, sample results less than 70 (or 10x7) would be qualified as not detected. In the case of the "5x" rule, sample results less than 35 (or 5x7) would be qualified as not detected.

Example 2: Sample result is less than the CRQL, and is also less than the 5x or 10x Multiple of the blank result.

	Rule	
	10x	5x
Blank Result	6	6
CRQL	5	5
Sample Result	4J	4J
Final Sample Result	5U	5U

Not that data are not reported as 4U, as this would be reported as a detection limit below the CRQL.

Example 3: Sample result is greater than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	10	10
CRQL	5	5
Sample Result	120	60
Final Sample Result	120	120

For both the "10x" and "5x" rules, sample results exceeded the adjusted blank results of 100 (or 10x10) and 50 (or 5x10), respectively, and therefore are not qualified.

VI . System Monitoring Compounds

A. Review Items: Form II VOA (From II LCV), quantization reports, and chromatograms.

B. Objective:

Laboratory performance on individual samples is established by means of spiking activities. All sample are spiked with system monitoring compounds, SMC, (formerly referred to as surrogates) just prior to sample purging. The evaluation of the results of these system monitoring compounds is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

C. Criteria:

1. Three system monitoring compounds (1,2-dichloroethane-d4, bromofluorobenzene, and toluene-d8) are added to all samples and blanks to measure their recovery in environmental samples in sample and blank matrices.

   (For data generated through the Low Concentration Water Method: A single system monitoring compound, bromofluorobenzene, is added to all samples and blanks to measure the recovery in sample and blank matrices)

2. Recoveries for system monitoring compounds in volatile samples and blanks must be within the limits specified in the Method.

D. Evaluation:

1. Check raw data (e.g., chromatograms and quantization reports) to verify the recoveries on the System Monitoring Compound Recovery Form - Form II VOA (Form II LCV). Check for any calculation or transcription errors.

2. Check that the system monitoring compound recoveries were calculated correctly. The equation can be found in the Method.

3. The following should be determined from the System Monitoring Compound Recovery form(s):

V.Blanks

A. Review Items: Form I SV-1 and SV-2 (Form 1 LCSV-1 and LCSV -2), Form IV SV (Form IV LCSV), chromatograms, and quantization reports.

B. Objective:

The purpose of laboratory (or field) blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, trip blanks, and equipment blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blanks.

2. The method blank must be analyzed on each GC/MS system used to analyze that specific group or set of samples.

D. Evaluation:

1. Review the results of all associated blank, Form I SV-1 and SV-2, and raw data (chromatograms and quantization reports) to evaluate the presence of target and non-target compounds in the blanks.

2. Verify that a method blank analysis has been reported per matrix, per concentration level, for each extraction batch and for each GC/MS system used to analyze semivolatile samples. The reviewer can use the method blank summary (Form IV SV) to assist in identifying samples associated with each method blank.

E. Action:

If the appropriate blanks were not analyzed with the frequency described above, then the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may to obtain additional information from the laboratory. The situation should be noted for TPO action.

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the common phthalate contaminants, or 5 times the amount for other compounds. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value.

Specific actions are as follows:

6. If a volatile compound is found in a blank but not found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action.

7. Any semivolatile compound detected in the sample (other than the common volatile laboratory contaminants), that was also detected in any associated blank, is qualified if the sample concentration is less than five times (5x) the blank concentration. The quantization limit may also be elevated. Typically, the sample CRQL is elevated to the concentration found in the sample. The reviewer should use professional judgment to determine if further elevation of the CRQL is required. For phthalate contaminants, the results are qualified "U" by elevating the quantization limit to the sample concentration when the sample result is less than 10 x the blank concentration.

   The reviewer should note that blanks may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" and "10x" criteria, such that a comparison of the total amount of contamination is actually made.

   Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. However, if the reviewer determines that the concentration is from a source other than the sample, he/she should qualify the data. In this case, the "5x" or "10x" rules may not apply; the

sample value should be reported as a non-detect. An explanation of the rationale used for this determination should be provided in the narrative accompanying the Regional Data Assessment Summary.

3. If gross contamination exists (i.e., saturated by GC/MS), all affected compounds in the associated samples should be qualified as unusable(R) due to interference.
This should be noted for TPO action if the contamination is suspected of having an effect on the sample result.

4. If inordinate number of other target compounds are found at low levels in the blank(s), it may be indicative of a problem and should be noted for TPO action.

5. The same consideration given to the target compounds should also be given to Tentatively Identified Compounds (TICs), which are found in both the sample and associated blank(s). (See SV Section XIII for TIC guidance.)

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Example 1: Sample result is greater than the Contract Required Quantization Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	12	12
CRQL	10	10
Sample Result	50	40
Qualified Sample Result	50U	40U

In the example for the “10x” rule, sample results less than 120 (or 10x12) would be qualified as non- detected. In the case of the “5x” rule, sample results less than 60 (or 5x12) would be qualified as non-detects.

Example 2: Sample result is less than the CRQL, and is also less than the 5x or 10x Multiple of the blank result.

	Rule	
	10x	5x
Blank Result	12	12
CRQL	10	10
Sample Result	8J	8J
Qualified Sample Result	10U	10U

Not that data are not reported as 8U, as this would be reported as a detection limit below the CRQL.

Example 3: Sample result is greater than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	15	15
CRQL	10	10
Sample Result	160	80
Qualified Sample Result	160	160

For both the “10x” and “5x” rules, sample results exceeded the adjusted blank results of 150 (or 10x15) and 75 (or 5x15), respectively, and therefore are not qualified.

VI. Surrogate Spikes

F. Review Items: Form II SV-1 and SV-2 (Form II LCSV), chromatograms, and quantitation reports.

B. Laboratory performance on individual samples is established by means of spiking activities. All sample are spiked with surrogate compounds, prior to sample preparation. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside

the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

C. Criteria:

1. Surrogate spikes, 4 acid compounds (3 required and 1 advisory) and 4 base/neutral compounds (3 required and 1 advisory) are added to all samples and blanks to measure their recovery in sample and blank matrices.
(*For data generated through the Low Concentration Method: Surrogate spikes, 3 acid compounds and 3 base/neutral compounds, are added to all samples and blanks to measure their recovery in sample and blank matrices.)*

2. Surrogate spike recoveries for semivolatile samples and blanks must be within the limits specified on in the SOW and on Form II SV-1 and SV-2.

(*For data generated through the Low Concentration Method: Surrogate spike recoveries for semivolatile samples and blanks must be within the limits specified in the method and on Form II LCSV)*

D. Evaluation:

5. Check raw data (e.g., chromatograms and quantitation reports) to verify the surrogate spike recoveries on the Surrogate Recovery Form II SV-1 and SV-2 (Form II LCSV). Check for any transcription or calculation errors.

6. Check that the surrogate spike recoveries were calculated correctly. The equation can be found in the method.